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Donald J. Burton

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Novel, general methods for the preparation of thermally stable perfluorinated organometallic reagents were developed. F-vinyl iodides were prepared as precursors to F-vinyl organometallics. A variety of polyfluorinated cadmium, zinc, and copper reagents were developed as synthetic reagents for the introduction of polyfluorinated alkyl, aryl, and allyl groups. SET chemistry was developed for the regiospecific addition of iodofluoroacetates and iodofluoromethylphosphonates to functionalized alkenes, and to accomplish a useful preparation of allylsulfonyldifluoroacetates and acetamides. Alkylation reactions and acylation reactions of <-fluorocarboxy phoshorus ylides were developed as a useful entry to precursors which could be easily hydrolyzed to X-fluoro ester and  $\propto$ -fluoro- $\beta$ -keto esters.

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  Department of Chemistry
  University of Iowa

University of Iowa Iowa City, Iowa

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Dr. Haridasan Nair Dr. Zai-Ming Qiu

Dr. Zhen-Yu Yang

Visiting Research Scientists:

Y. Tarumi

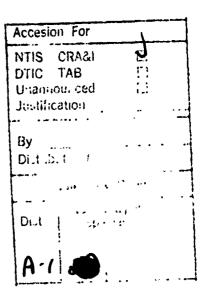
V. Tortelli

M. Yamamoto



Charles R. Davis H.J. Tsai Hengyao Lu Y. Wang B.V. Nguyen L. Xue







## 8. Publications:

"Generation of Trifluoromethylcopper from Chlorodifluoroacetate"; J.G. MacNeil, Jr. and D.J. Burton, J. Fluorine Chem., 1992, in press.

"Fluorinated Organometallics: Perfluoroalkyl and Functionalized Perfluoroalkyl Organometallic Reagents in Organic Synthesis"; Z-Y. Yang and D.J. Burton, Tetrahedron Reports, 1992, in press.

"Perfluoroisopropylcadmium and Copper: Preparation, Stability and Reactivity"; H.K. Nair and D.J. Burton, J. Fluorine Chem., 1992, in press.

"A Novel, General Method for the Preparation of α,α-Difluoro Functionalized Phosphonates"; Z-Y. Yang and D.J. Burton, Tetrahedron Lett., 32 (8), 1019 (1991).

"New Approaches to  $\alpha$ -Fluoro and  $\alpha,\alpha$ -Difluoro Functionalized Esters"; D.J. Burton, A. Thenappan and Z-Y. Yang, ACS Symposium Series #456, "Selective Fluorination in Organic and Bioorganic Chemistry", John Welch, Editor, Chapter 7, 91, 1991.

"A New Approach to  $\alpha,\alpha$ -Difluoro-Functionalized Esters"; Z-Y. Yang and D.J. Burton, J. Org. Chem., <u>56</u>, 5125 (1991).

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"Preparation of Ethyl 2-Fluoroacrylate, CH<sub>2</sub>=CFCOOEt"; A. Thenappan, D.J. Burton, J. Fluorine Chem., <u>48</u> 153 (1990).

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- "Copper Catalyzed Addition Reaction of Iododifluoroacetates To Olefins"; Z-Y. Yang and D.J. Burton, J. Fluorine Chem., 45, 435 (1989).
- "A Facile Preparation of <u>Gem</u>-Difluorohomoallylic Alcohols"; Z-Y. Yang and D.J. Burton, J. Fluorine Chem., <u>44</u>, 339 (1989).
- "Preparation of <u>E</u>-1,2,3,3,3-Pentafluoropropene; <u>Z</u>-1,2,3,3,3-Pentafluoropropene, and <u>E</u>-1-Iodopentafluoropropene"; D.J. Burton, T.D. Spawn, P.L. Heinze, A.R. Bailey, and S. Shin-ya, J. Fluorine Chem., <u>44</u>, 167 (1989).
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- "A Facile Preparation of Ethyl  $\alpha$ -Fluoroalkanoates"; A. Thenappan and D.J. Burton, Tetrahedron Lett., 30 (28), 3641 (1989).
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## 9. ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

The objectives of this project were: (a) to develop new, novel, stereospecific general methods for the preparation of thermally stable polyfluorinated organometallic reagents. An adjunct of this strategy was to elucidate the structure of these reagents and to develop their application particularly for the synthesis of functionalized fluorocarbon derivatives; (b) to utilize commercially available precursors (where applicable) as the source of fluorine in reactive intermediates in order to facilitate utilization of the synthetic methodology by other workers and to assist scale up of the preparative procedures; (c) to develop single electron transfer (SET) methodology as a useful, easily scaled-up synthesis of functionalized fluorine-containing compounds; (d) to utilize fluorine-containing ylides as synthetic intermediates in the synthesis of  $\alpha$ -fluoro- $\alpha$ , $\beta$ -unsaturated esters; and (e) to develop Pd(0) catalyzed processes for the preparation of perfluoro vinyl or polyfluoroaryl functionalized derivatives.

Initial efforts were directed to the stereospecific preparation of Evinyl iodides as precursors to E-vinyl organometallics. A successful approach to E-1-iodopentafluoropropene was developed from hexafluoropropene (HFP) via reaction of HFP with tertiary phosphines to give the Z-perfluoropropenyl phosphorane. Subsequent hydrolysis of the vinyl phosphorane gave E-1,2,3,3,3-Pentafluoropropene. Treatment of this E-1-hydro-F-propene with SbF<sub>5</sub> gave Z-1,2,3,3,3-Pentafluoropropene. Subsequent metallation of the Z-1-hydro-F-propene (with n-BuLi) and iodination of the organic lithium intermediate gave pure E-1-Iodopentafluoropropene. Trifluorovinyl iodide and both E- and Z-1iodopentafluoropropenes are stereospecifically converted to the corresponding E-vinyl zinc reagents. These E-vinyl iodides readily participate in Pd(0) catalyzed reactions; with terminal alkynes, we have developed a general stereospecific route to fluorinated envnes. fluorinated aryl iodides undergo Pd(0) catalyzed coupling reactions with 1alkynes and this methodology provides a high yield, easily scaled-up entry to polyfluorinated aryl alkynes and di-ynes.

A variety of polyfluorinated cadmium, zinc and copper reagents have been developed as synthetic reagents for the introduction of polyfluorinated alkyl, allyl and aryl groups. Perfluoroisopropylcadmium and copper were prepared and the stability and reactivity of these secondary perfluoroallyl organometallics was investigated. A new, novel generation of trifluoromethylcopper was developed based on the cheap

precursor, chlorodifluoroacetate. It was also unequivocally demonstrated that trifluoromethylcopper(I) could be cleanly oxidized to a stable trifluoromethylcopper(III) and the first example of a perfluoroalkylcopper(III) complex was prepared and its structure demonstrated by x-ray crystallography. The first preparation of perfluoroallyl cadmium and copper reagents was developed and applications of these novel F-allylic organometallics were demonstrated. Similarly, the facile preparation of E-phenyl Mono- or Bis-cadmium and copper reagents was developed from the commercially available bromo- or iodopentafluorobenzene and 1,2-dibromotetrafluorobenzene, and the application of these novel reagents for the synthesis of functionalized E-The first practical preparation of aryl derivatives was demonstrated. perfluoroalkyl allenes and difluoromethyl allenes was developed from the reaction of perfluoroalkyl copper and difluoromethyl copper reagents with propargyl chlorides and tosylates. In contrast to previous reports, this methodology provided a safe and facile route to these perfluorinated building blocks. The functionalized organometallic, (RO)<sub>2</sub>P(O)CF<sub>2</sub>ZnBr, was developed as a useful stable organometallic reagent and could be utilized in the first, high yield practical synthesis of the novel fluorinated chelating agent,  $(RO)_2P(O)CF=CFP(O)(OR)_2$  (where R=H, Et). This reagent was also utilized in regiospecific allylation reactions and provided a useful synthetic route to 1,1-difluoro-3-alkenephosphonates. The trifluorovinyl copper reagent was utilized for the development of diene precursors which could be reacted with sulfur trioxide (sulfonation) to give novel fluorinated βsultones. Ring opening of these sultones gave novel fluorinated sulphonyl fluorides useful in polymerization studies. The commercially available olefin, 3-bromo-3,3-difluoropropene, was utilized in the metal mediated regiospecific gem-difluoroallylation of aldehydes and ketones as an entry to  $\alpha, \alpha$ -difluorohomoallylic alcohols. The regiospecificity was correlated with theoretical studies of the 1,1-difluoroallylic anion.

SET chemistry was developed for the regiospecific addition of iododifluoroacetates and iododifluoromethylphosphonates to functionalized alkenes. Reduction of the adducts with a new Ni $^{\circ}$  catalyst provided a practical synthetic route to  $\alpha,\alpha$ -difluoro-functionalized esters and  $\alpha,\alpha$ -difluoro-functionalized phosphonates. The SET methodology avoided the use of expensive, toxic and hazardous reagents and provided the first practical, easily scaled-up, safe route to these important fluorinated precursors. Similar strategy was utilized to accomplish a useful preparation of allylsulphonyldifluoroacetates and acetamides. A combination of organometallic chemistry and SET chemistry was employed in the preparation of the mixed acid, (HO)<sub>2</sub>P(O)CF<sub>2</sub>SO<sub>3</sub>H.

Alkylation and acylation reactions of  $\alpha$ -fluorocarboalkoxy phosphorus ylides (phosphonium and phosphonate) were developed as a useful entry to precursors, which could be readily hydrolyzed (aqueous NaHCO3) to  $\alpha$ -fluoro ester and  $\alpha$ -fluoro- $\beta$ -keto esters. An adjunct of this strategy resulted in the development of a reduction olefination reaction of fluorinated esters. This methodology provided an efficient synthesis of  $\alpha$ -fluoro- $\alpha$ ,  $\beta$ -unsaturated esters directly from polyfluorinated esters. Thus, the necessity to prepare the easily hydrated and easily polymerized pr , fluorinated aldehydes was avoided. This methodology was utilized to develop a practical, large scale synthesis of the monomer, ethyl-2-fluoroacrylate.

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The Preparation and Reaction of	Fluorinated Phe	enyl Mono- or	Bis-Cadmin	um and	Copper Reagents
Burton, D.J., Yang, Zhen-Yu; Ma	cNeil, Kathryn J	J.			
13a, TYPE OF REPORT 13b TIME ( Reprint/Final FROM_		14. DATE OF REPO	RT (Year, Mont	n, Day)	15. PAGE COUNT 5
16. SUPPLEMENTARY NOTATION	<u> </u>	<del></del>			
J. Fluorine Chemistry 52, 251-2	55 (1991)				
17. COSATI CODES	18 SUBJECT TERMS	(Continue on reven	se if necessary a	nd identii	y by block number)
FIELD GROUP SUB-GROUP	] fluorinated o	organometalli	cs; aromat:	ic fluo	rine compounds;
					nated aryl cadmi
	reagents: flu	uorinated arv			
19. ABSTRACT (Continue on reverse if necessar				DVC	<b></b>
Tetrafluorodibromobenzenes reac ranging from 25°C to 60°C to					
Treatment of the mono- cadmium	give promotetra	r mith waste	cadmium rea;	t 1000 t 1000	n excertent yie Lafforde the te
fluorophenylbiscadmium reagents	. The mono- and	d bis-conner	reagente a	re obta	ined via metath
of the corresponding cadmium re	agents with cur	rous bromide	at room te	mperatu	ire. Allylation
acylation of the biscopper reag	ents give the co	orresponding	allylated	and acy	lated tetrafluo
benzenes, respectively.		. 0	•	•	
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228. NAME OF RESPONSIBLE INDIVIDUAL			(include Area C	ode)   22c	. OFFICE SYMBOL
Dr. Fred Hedberg		202-767-49			NC.

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11. TITLE (Include Security Classification) Palladium/cuprous Iodide Cat	talyzed Coupling	of Substitut	ed Tetraflu	oropheny1	
Halides with 1-alkynes					
12. PERSONAL AUTHOR(S) Ba V. Nguyen, Z.Y. Yang and	D.J. Burton				
13a. TYPE OF REPORT 13b. TIME	COVERED	14. DATE OF REPOR	RT (Year, Month.	h, Day) 15. PAGE	COUNT
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16. SUPPLEMENTARY NOTATION  J. Fluorine Chemistry 50,	265-70 (1990)				
17. COSATI CODES	18. SUBJECT TERMS (	Continue on rever	e if necessary so	d identify by Na	ck number)
FIELD GROUP SUB-GROUP	fluorinated or	rganometallic	cs. fluorina	ated acetyle	enes.
	halogenated al	lkynes, fluor	rinated aron	matics, pal	ladium
19. ABSTRACT (Continue on reverse if necessal	ary and identify by block n	number)			
In the presence of cup substituted tetrafluorophen alkynes in good to excellen N.N-dimethylaming, morpholi	prous iodide, the nyl halides with l nt yields under mi ino) tetrafluoroph	palladium ca l-alkynes giv aild condition ahenyl iodides	ves the corr ns. Both 4- s and bromic	responding 1 I-substituted ides work we	fluoroaryled (methoxy, ell, and
alkyl, trimethylsilyl, phen are tolerated under the rea synthesis of substituted fl	nyl, hydroxy, and action conditions.	l ether functi . This metho	ionalities i	in the alkyl	ne motety
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11. TITLE (Include Security Classification)		مر <u>د را در </u>			
The Preparation and Allylati	on of Perfluoroal	lyl Cadmium	and Copper	Reagents	
12. PERSONAL AUTHOR(S)			····		
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J. Fluorine Chemistry, 50, 2	57-263 (1990)				
17. COSATI CODES	18. SUBJECT TERMS				
FIELD GROUP SUB-GROUP	fluorinated or and copper real	rganometalli agents, perf	cs, fluorii luoroallyl	nated ally halides.	r cadmium perfluoro-
	allylation rea	actions			
19. ABSTRACT (Continue on reverse if necess Perfluoroallyl iodide reacts the F-allylcadmium reagent. the F-allylcopper reagent. hexadiene. Only a low yield allylcopper react with allyl	readily with acid Metathesis of F-With zinc powder, of F-allylzinc wa	d-washed cad allylcadmium perfluoroal as detected.	with Cu(I) lyl iodide Both F-a	)Br at -35 affords m llvlcadmiu	O C in DMF giver aim and F-1,5-
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1. TITLE (Include Security Classification)					_	_
Sulfonation of [2,3-Dichloropr S-Sultone and Derivatives	opyljtrifluoroeti	nylene: Synt	hesis of a l	iew Fl	uorinat	ed
2. PERSONAL AUTHOR(S)		<del></del>				
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J. Fluorine Chemistry, <u>50</u> , 31	<b>-</b> 46 (1990)					
7. COSATI CODES	18 SUBJECT TERMS	(Continue on revers	e if necessary and	identify	by block	number)
FIELD GROUP SUB-GROUP	fluoroolefins	; fluorinat	ed sultones;	flu	orinate	d acids:
	fluorinated a			•		<b>-</b>
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19. ABSTRACT (Continue on reverse if necessary					_	
Two new olefins, CH <sub>2</sub> BrCHBrCH <sub>2</sub>	Cr=Cr2 and Ch2C10	HCICH2CF=CF2	, have been	prepa	red as	precursors
to fluoro $\beta$ -sultones. The new	fluorinated sulf	one, CH <sub>2</sub> C1CH	C1CH <sub>2</sub> CFCF <sub>2</sub> OS	30 <sub>2</sub> , w	as obta	ined (from
the sulfonation of CH <sub>2</sub> C1CHC1CH	2CF=CF2), along v	vith its rear	ranged isome	er,		
CH <sub>2</sub> C1CHC1CH <sub>2</sub> CF(SO <sub>2</sub> F)C(O)F, and	nyarolysis produ	ice, Ch <sub>2</sub> CICHC	ICH <sub>2</sub> CF(SO <sub>2</sub> F)	C(0)0	н.	
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20332-6448  11. TITLE (Include Security Classification) Synthesis and Characterization of (E) and (Z)-1,2-Difluoroethenediyl Bis Phosphonates  12. PERSONAL AUTHOR(S) L.G. Sprague, D.J. Burton, R.D. Guneratne and W.E. Bennett  13a. TYPE OF REPORT   13b. TIME COVERED   14. DATE OF REPORT (Year, Month, Day)   15. PAGE COUNT   11   16. SUPPLEMENTARY NOTATION  J. Fluorine Chemistry   49, 75-85 (1990)  17. COSATI CODES   18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if necessary and identify by block number)   19. ABSTRACT (Continue on reverse if neces	REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188
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20332-6448 11. TITLE (Include Security Classification)		61102F	<u> </u>			
Preparation of Ethyl 2-Fluore	oacrylate, H <sub>2</sub> C=0	CFCO <sub>2</sub> Et				
12. PERSONAL AUTHOR(S) A. Thenappan and D.J. Burton						
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17. COSATI CODES	18. SUBJECT TERMS		•	-	-	
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19. ABSTRACT (Continue on reverse if necessary	phosphonate a					
From ethyl formate, a l developed. This work provid of this monomer.						
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17.	COSATI	CODES			(Continue on reven				
FIELD	GROUP	SU	B-GROUP		sters, fluor				
·	<u> </u>		<del></del>	fluorinated a	arbanions, u Idehydes	nsaturated t	luor	nated	esters,
19. ABSTRACT	(Continue on	revers	e if necessar	y and identify by block					
А	reduction-	olef	ination	sequence has be	en used to c	onvert ester	's to	a-fluc	ro α,3-
unsatur	ated ester	S.	in the p	presence of diis with [(EtO) <sub>2</sub> P <u>(</u> (	SOBUTYlalumin NCEC(G\OE+l-	um hydride,	este	ers are	reduced to
good vi	elds with	hiah	stereos	selectivity. The	ne reaction i	s applicable	to	aliphat	ic. aro-
matic,	cyclic, u	isatu	rated, p	perfluorinated,	and partiall	y fluorinate	ed es	ters.	The E/Z rati
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mixture				little influence					
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17. COSATI CODES		18. SUBJECT TERMS					
FIELD GROUP SUB-GROUP Fluorinated copper reagents, fluorinated organ				c3, 114011III	u ceu ·	or garrom	c tu , , , c , ,
			a comband				
19. ABSTRACT (Continue on reverse							
Perfluoroalkyl co	pper rea	gents react wit	h propargyl	halides or	tosyl	ates in	UMF. or
	pper rea	gents react wit	h propargyl	halides or ifically in	tosyl good	ates in yield.	UMF. or
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20332-6448 11. TITLE (Include Security Classification)		61102F		<u> </u>	
A Facile, General Method for t	he Preparation o	f Fluorinate	d Enynes		
12. PERSOMAL AUTHOR(S)					
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16. SUPPLEMENTARY NOTATION					
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17. COSATI CODES	18. SUBJECT TERMS (	Continue on reven	se if necessary and	didentify	by block number)
FIELD GROUP SUB-GROUP					ns; palladium
	catalysis; f	luorinated v	rinyl halide	s; co	upling reactions
19. ABSTRACT (Continue on reverse if necessor	y and identify by block n	umber)			
Fluorinated vinyl iodides, R <sup>1</sup> C			( <sup>i</sup> PrO) <sub>2</sub> P(O)	], cou	ple directly
with l-alkynes in the presence	of palladium an	d cuprous id	odide in tri	ethyla	mine to give
excellent yields of the fluori	nated enynes.				
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University of Iowa Iowa City, Iowa 52242		Bldg. 410 Bolling AFB, D.C. 20332-6448  9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER  AFOSR-89-0134  10. SOURCE OF FUNDING NUMBERS  PROGRAM PROJECT TASK WORK UNIT ACCESSION NO. 61102F				
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Alkylation of (Fluorocarbetho α-Fluoroalkanoates	xymethylene)trl	-u-outytpnos	pnorane: A	racile	Entry to	
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17. COSATI CODES	18. SUBJECT TERMS	Continue on revers	e if necessary and	d identify	by block number)	
FIELD GROUP SUB-GROUP	α-fluoroester α-fluorophosp		ed ylides;	alkyla	cion;	
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19. ABSTRACT (Continue on reverse if necessary	and identify by block r	number)	<del></del>			
(Fluorocarbethoxymethyl)tria acetate and tertiary phosphi						
phosphoranes. Reaction of t						
phorane with primary alkyl i						
hydrolysis of the alkylated						
In the case of secondary alk						
being decomposition of the p						
(fluorocarbethoxymethyl)phos	-			J 4		
the corresponding alkylated moiety either by base-induce						
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Synthesis and X-ray Structure A Remarkably Stable Perfluoroa			N-diethyldit	hiocar	bamato)copper;
12. PERSONAL AUTHOR(S)	ik, icopper (III)	Complex		<del></del>	
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J,. Chemical Society, Chemical	. Communications	s 1633-1634	(1989)		
17., COSATI CODES	18. SUBJECT TERMS (C	Continue on revers	e if necessary and	identify	by block number)
FIELD GROUP SUB-GROUP			Copper (III)	Compl	Lexes;
	trridoromet	thyl organom	erairics		;
19. ABSTRACT (Continue on reverse if necessary a					
The reaction between trifluoro	omethylcadmium i	reagent and	Br <sub>2</sub> Cu(edtc)	(edtc	= N, N-diethyl-
dithiocarbamato) or $CdI^{+}[(CF_{3})]$ yields the stable $Cu^{III}$ perflu	oroalkylcopper	complex. (C	FalaCuSC(S)N	Eta.	namide/ at -50 c
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Copper C	atalyzed A	ddition Reac	tion of Iododifl	Luoroacetate	s to Olefin	s	
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17.	COSATI			18. SUBJECT TERMS					
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11. TITLE (Include Security Classification) Preparation of E-1,2,3,3,3-P	entafluoropropene	<u>z</u> -1,2,3,3,	3-Pentafluo	roprop	ene and	
E-1-Iodopentafluoropropene						
12. PERSONAL AUTHOR(S) D.J. Burton, T.D. Spawn, P.L	. Heinze, A.R. Ba	iley and S.	Shin-Ya			
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Journal of Fluorine Chemistr	y, "Fluorine Chem	istry Synthe	sis", <u>44</u> 10	67-174	(1989)	
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19. ABSTRACT (Continue on reverse if necessary (Sulfodifluoromethyl) phosphonic time. This mixed phosphonic had been synthesized via oxida The sulfinate salt was prepare [(C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> F(O)CF <sub>2</sub> SO <sub>2</sub> ] <sub>2</sub> Cd precur	ic acid, (HO) <sub>2</sub> P( sulfonic acid wa ution of the cor ed from (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub>	0)CF <sub>2</sub> SO <sub>3</sub> H, h is prepared fi responding s	rom (C <sub>2</sub> H <sub>5</sub> O) ulfinate s	) <sub>2</sub> 2(0)C2 alt, (C <sub>2</sub>	2503Na, which	

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and all of the observed bands are similar to those of other the variations can be explaine the C-X bond. Several bands, sequence structure due to excipant the control of t	CF3CCX species, d without change particularly V <sub>1</sub> ted levels of V	and even tho s in force c and combina 10, the C-C-  21. ABSTRACT S Unclass  225. TELEPHONE	ugh the Arons of tions with C skeletal  ECURITY CLASSIFIED (Include Area Co	modes ther that V <sub>1</sub> , sho bend.	are less regular, an those involving ow pronounced  OFFICE SYMBOL	
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